AFRL-AFOSR-VA-TR-2016-0225

Printable Integrated Photonic Devices

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06/16/2016
Final Report

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Air Force Research Laboratory
AF Office Of Scientific Research (AFOSR)/RTA1

Arlington, Virginia 22203
Air Force Materiel Command

REPORT DOCUMENTATION PAGE	*Form Approved* *OMB No. 0704-0188*

1. REPORT DATE *(DD-MM-YYYY)*	**2. REPORT TYPE**	**3. DATES COVERED** *(From - To)*
04/05/2016	Final Report	March 1, 2012 - November 30, 2012

4. TITLE AND SUBTITLE
Printable Integrated Photonic Devices

5a. CONTRACT NUMBER
FA9550-14C-0020

5b. GRANT NUMBER
AF11B-T05-0103

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
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5d. PROJECT NUMBER

5e. TASK NUMBER
Final, Phase II

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
ABEAM TECHNOLOGIES INC.
5286 DUNNIGAN CT
CASTRO VALLEY CA 94546-1612

8. PERFORMING ORGANIZATION REPORT NUMBER
DUNS 14157437

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
USAF, AFRL
AF OFFICE OF SCIENTIFIC RESEARCH
875 N. RANDOLPH ST. ROOM 3112
ARLINGTON VA 22203

10. SPONSOR/MONITOR'S ACRONYM(S)
AFOSR/PKR3

11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release

13. SUPPLEMENTARY NOTES
Prepared in cooperation with Lawrence Berkeley National Laboratory

14. ABSTRACT
The objective of this US Air Force Phase II STTR project was to demonstrate imprintable, high refractive index material, high resolution imprint technology as well as imprinted photonic devices. A novel nanoimprint resist with refractive index over 2.0 and high optical transparency was synthesized. Novel "active" imprintable material was also developed with embedded QDs (optical gain media). An imprint process was developed that demonstrated unmatched sub-10 nm patterning resolution, high uniformity over a large area, and zero shrinkage. The developed technology is suitable for low cost fabrication of passive and active integrated optical devices such as nanolasers.

15. SUBJECT TERMS
STTR Report

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**			K. Munechika
					19b. TELEPHONE NUMBER *(Include area code)*
n/a	n/a	n/a	n/a		(360) 402-4112

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PRINTABLE INTEGRATED PHOTONIC DEVICES

STTR project Phase II - Status Report V
Jan 9th 2014 –Jan 8th 2016

Abstract:

A novel low-cost printing technology for manufacturing passive and active printable photonic components were developed based on nanoimprint lithography. Functional imprint materials were developed as well as the process to achieve state-of-the-art imprint processes with nearly zero-shrinkage in all dimensions and down to 10 nm resolution. A revolutionary type of imprint resist integrated with colloidal quantum dots (QDs) as optical gain media were developed for printing ***active*** photonic devices. Several types of printable photonic devices were successfully demonstrated including micro lens array, light extraction layer on the commercial LEDs, and revolutionary printable nanocavies for low-cost and ultra-small nanolasers.

<u>Main Accomplishments of the Phase II efforts can be summarized as following:</u>

1. Developed printable high refractive index functional materials and processes for low cost imprinting of photonic devices
2. Development of active nanoimprint resist integrated with colloidal QDs
3. Printed Photonic Device 1:
   a. Demonstrated single step imprinting of photonic crystals with QDs with enhanced light intensity
   b. Demonstrated working nanocavities for nanolasers
4. Printed Photonic Device 2: Demonstrated high refractive index light extraction layer integrated with commercial LEDs (prototype 1)
5. Printed Photonic Device 3: Optically validated micro lens array imprinted with high refractive index materials (prototype 2)

I- Development of Functional Materials and Imprint process

- **Development of functional high refractive imprint resist**

To fabricate photonic devices for visible light, a material with both a high refractive index (n) and low extinction coefficient (k) in the visible range is required. High refractive index ensures tighter confinement of the optical mode in the patterned medium, and a smaller footprint, while a low extinction coefficient is required to prevent optical absorption and losses when the light travels through the material. One of the most promising materials for this purpose is titanium dioxide (TiO_2), having n>2 and an excellent optical transmission (>90%) down to 400 nm wavelength. We developed a hybrid organic-inorganic, imprintable high refractive index (n =1.9) and high transmittance suitable specifically for printable devices in the entire visible region. (Figure 1)

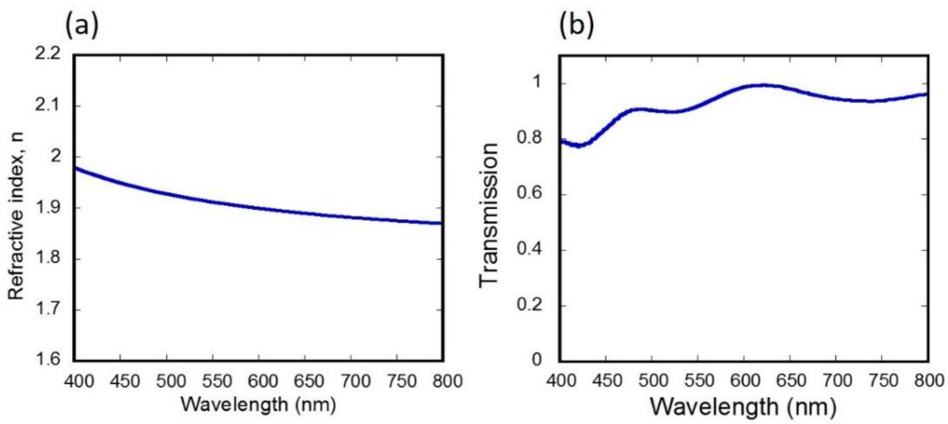

Figure 1: aBeam's proprietary TiO2 (ceramic) based high-refractive index imprint material (a) refractive index vs. wavelength, and (b) transmittance vs. wavelength.

- **Development of active functional resist integrated with colloidal quantum dots (QDs)**

The main purpose of the project was to develop technology for integrated photonic devices. To this regard, we have developed a revolutionary functional imprint resist which are integrated with colloidal quantum dots (optical gain medium). QDs exhibit strong absorption and band-edge emission whose frequency is dependent on the size and shape, which provides a unique opportunity to tune the frequency of photoluminescence, or, in other words, the color of the working device. In order to ensure the optical properties of QDs are maintained after incorporation and imprinting, thermally stable CdSe/CdS core/shell quantum dot nanocrystals were synthesized and incorporated into the sol-gel precursor. TiO_2 based imprint materials typically require high annealing temperature at which the optical performance of QDs are significantly compromised due to thermally activated non-radiative pathways. Maintaining the optical property at the processing condition is a key requirement for a functional device made by nanoimprinting. A specific colloidal chemistry was employed to synthesize thermally stable QDs. Various solvents were test to ensure uniform incorporation of QDs were confirmed. **This novel active printable material has recently attracted interests from numerous companies.**

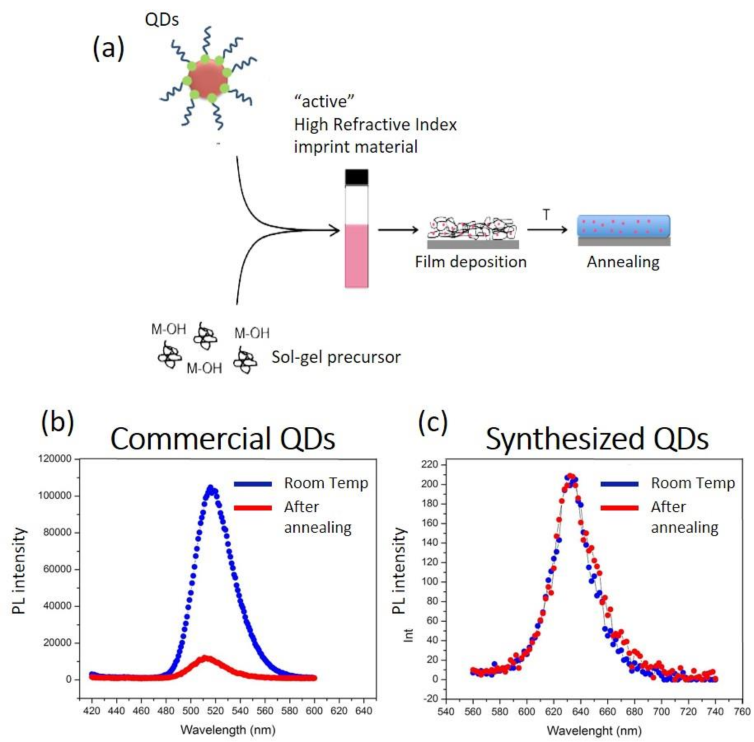

Figure 2: A scheme of incorporating colloidal quantum dots into sol-gel precursor to formulate active functional imprint material. Comparison of photoluminescence intensity between room temperature (blue) and after annealing (red) of commercial QDs (b) and thermally stable QDs (c).

- **Establishment of state of the art imprint process of high refractive index functional materials**

An imprint technology was developed and optimized for patterning titania films with high fidelity and unmatched resolution (Fig. 3 a-c). Reverse printing of inorganic resist was developed to drastically reduce the shrinkage down to ***zero in all directions*** (previously vertical shrinkage was approximately 40 %). While titania films are known to be difficult to imprint due to its mechanical properties, our process ensures the high quality of features at low edge roughness. To demonstrate a functioning photonic device, two dimensional photonic crystals were printed as depicted in Fig. 3(d)-(e). Optical properties were measured using custom built setup and results were compared to theoretical predictions of the same dimensions (hole diameter and pitch). The results were in excellent agreement validating the functionality of printed photonic devices. Another key improvement in the process was a made by lowering the annealing temperature during imprinting; previously, annealing at 400-500 C were required to achieve high refractive index and optical transparency. Our current process require only 180 C to achieve refractive index value of 1.9 with high optical transparency, thus, it opens up a wider range of applications where our imprint process can be integrated as a part of device fabrication procedure, where it was not possible, previously.

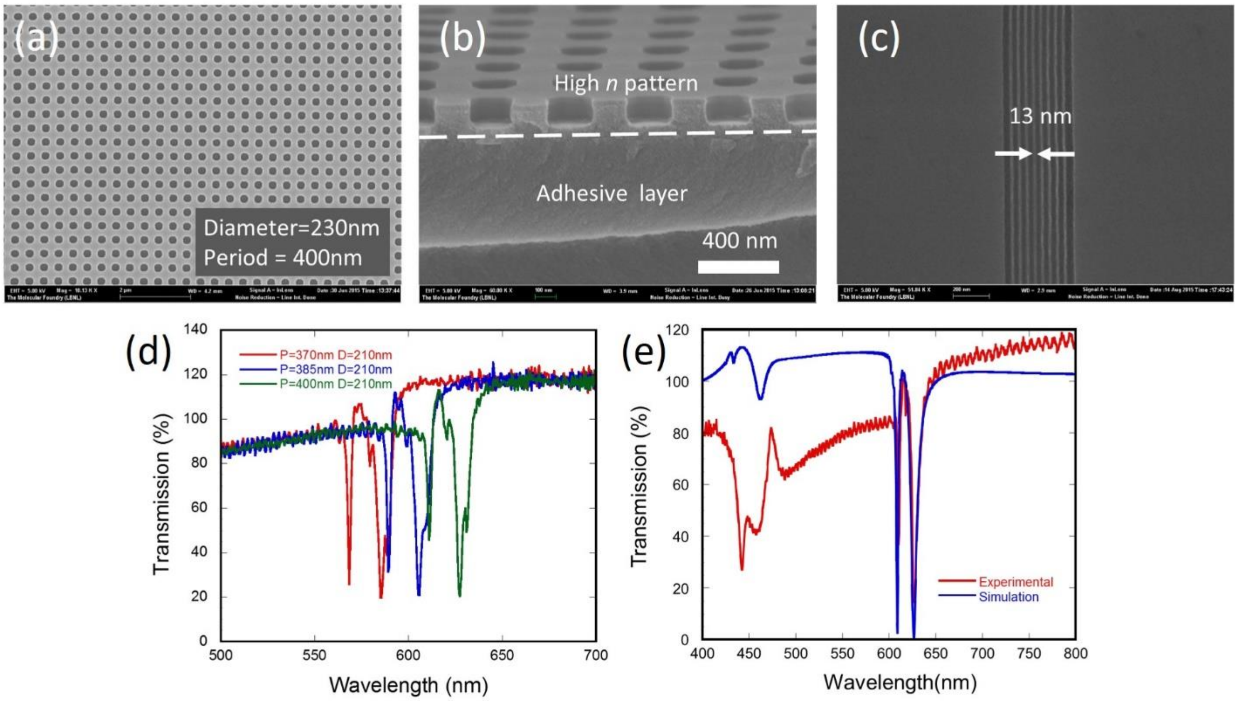

Figure 3: Demonstration of state-of-the-art imprinting process developed during the project. SEM images show TiO2 based functional materials can be imprinted with high-fidelity and high resolution (a)-(c)

In addition to the planned work and to show the versatility of our high refractive index imprintable polymer and our imprinting process, we have successfully demonstrated the imprinting of photonic crystals onto a flexible substrate, PET (polyethylene terephthalate), as shown in Figure 4, where an array of 500 two-dimensional photonic crystals were printed. Color variations observed across the patterned area (right picture) is related to different resonance frequency associated with each photonic crystal patterns with variable geometrical parameters. This is only achievable if the pattern transfer is successful with high fidelity across the entire substrate. **This demonstrates that our functional resist as well as imprint technology is suitable for printing flexible photonic devices.**

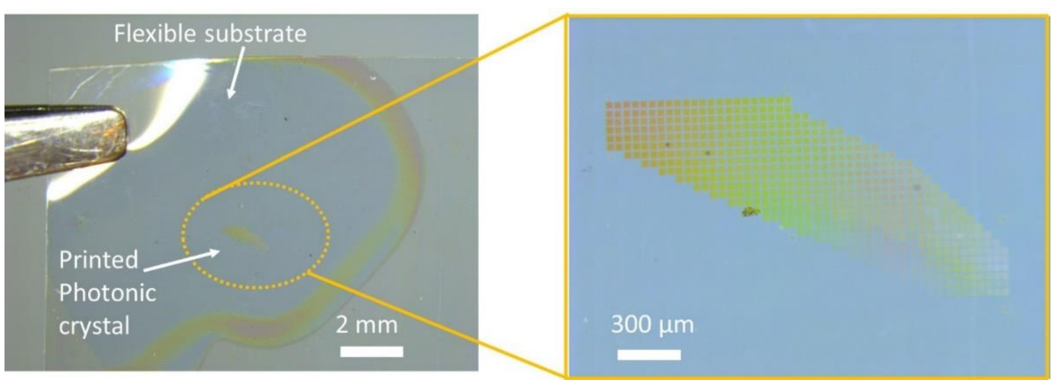

Figure 4: Flexible printed two dimensional photonic crystals. Each square represents a two dimensional photonic crystal.

II: Printed Photonic Device 1: Demonstration of single step imprinting of active photonic crystals with enhanced light intensity.

- **Demonstration of active two dimensional photonic crystals**

We have tested and characterized number of *active* two-dimensional photonic devices imprinted into our active functional resist. (Figure 5). Fluorescence intensities of QDs were compared between the regions with and without pattern within the same printed device. We measured nearly 10 fold increase in the fluorescence intensity from the patterned area. We demonstrated that the degree of photoluminescence enhancement is highly frequency dependent and the largest fluorescence enhancements comes from photonic crystals with the guided mode resonance frequency that spectrally overlaps with the excitation and emission frequency of the QDs used for this study (630 nm with FWHM 25 nm (Figure 5b). In contrast, when there is no spectral overlap, we do not observe any fluorescence enhancement (Figure 5c). This is a clear indication that the amplified fluorescence intensity is due to the presence of in-plane guided mode resonance: QDs are getting excited more efficiently inside the photonic crystal when the excitation wavelength matches the GMR. This represents a simple and an effective way to improve emission intensity of embedded QDs and represents a powerful and cost-effective route for the development of numerous nanophotonic structures and devices. ***This novel technology has attracted strong interest from a major display company.***

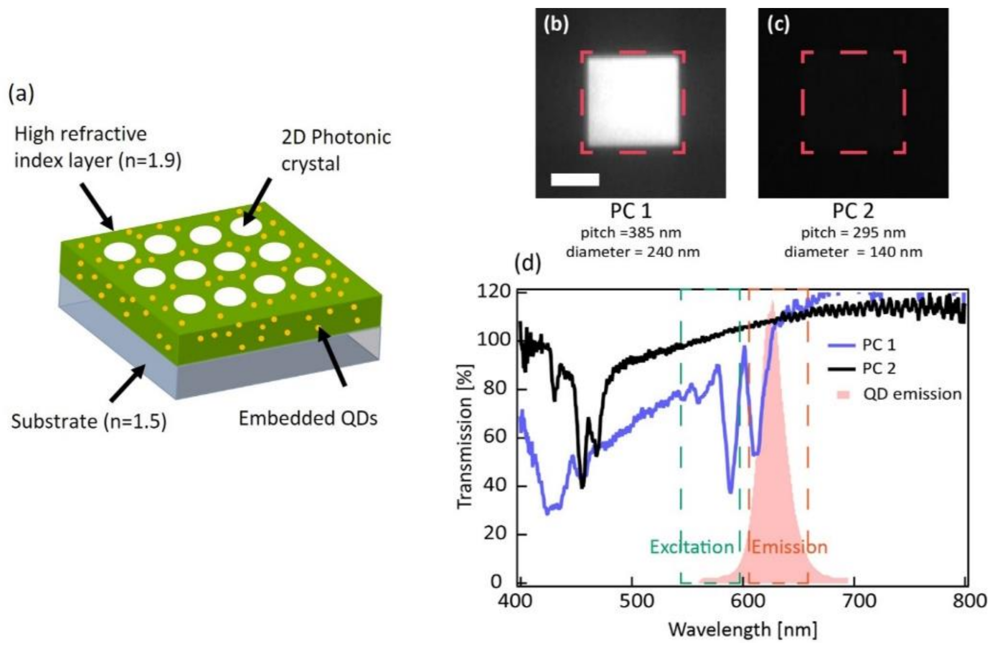

Figure 5: a) A scheme of a printed photonic device with embedded CdSe/CdS QDs. Fluorescence micrographs taken from different printed photonic crystals with QDs under the same illumination condition: b) PC 1 (pitch = 385 nm, diameter = 240 nm) and c) PC2 (pitch = 295 nm, diameter 140 nm). Red dashed lines highlight the location of the printed photonic crystals. d) Transmission spectra of PC1 and PC2. Green and Red dashed lines indicate the wavelengths used to excitation (green) and collection (red) of QD photoluminescence. Red shaded spectrum is the photoluminescence of the QDs used in this study. (Scale bar = 50 μm)

- **Simulation of one-dimensional photonic crystal cavity for nanolasers**

To fabricate novel printable photonic device such as nanolaser, one-dimensional cavity structure was designed using Finite Difference Time Domain simulation (FDTD) by Lumerical. The 1d cavity structure is shown in Figure 6. The main advantage of using 1d photonic crystal to create a cavity is that 1d photonic crystal has bandgap at any refractive index contrast. Relatively high refractive index contrast achievable using the material we have developed (n =1.9) provide benefit by reducing the cavity mode – which is a key to achieve a nanolaser. The simulated resonance mode profile is shown in 6b.

The decay rate of the quantum dot inside the cavity changes due to the change in the density of states. The simulation of the radiative decay rate inside 1d cavity, normalized to the decay rate of the quantum dot outside the cavity, is shown in Figure 6c. As one can see, the radiative decay rate has a narrow peak that corresponds to the cavity resonance wavelength. The modified decay rate can be observed by measuring the change in the PL spectrum. Modification of the decay rate is used to increase quantum dot efficiency, modify emission spectrum and create nanolasers.

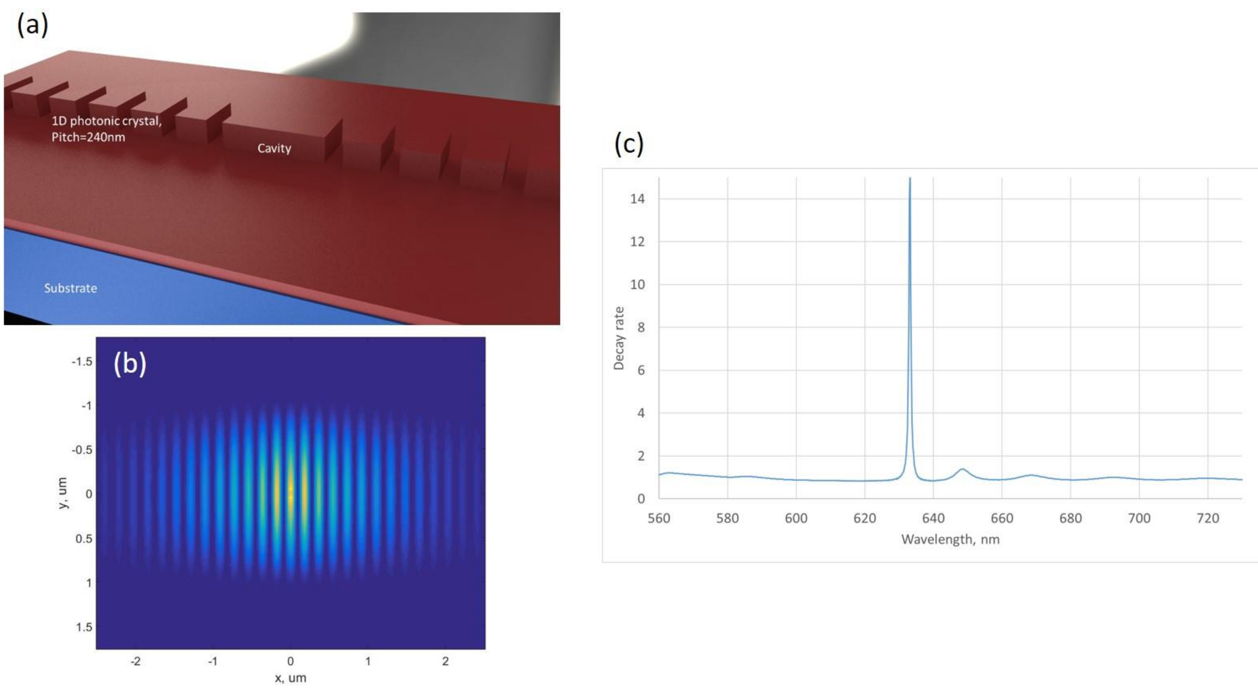

Figure 6 .a) Schematic illustation of 1 dimentional nanocavity. b) FDTD simulation of the cavity mode. (c) FDTD simulations of the decay rate of the quantum dot photoluminescence inside the cavity.

- **Demonstration of printed one-dimensional nanocavity for ultra-small laser**

Photonic crystal cavities samples were tested using custom built confocal microscope setup. An array of 1D cavities were printed with embedded optical gain medium. The quantum dots photoluminescence was excited using 470 nm picosecond laser pulses and measured using Andor CCD spectrometer. The typical results of the confocal scan measurements are shown in Figure 7 a-b. As one can see from the results, the presence of the cavity shifts the photoluminescence spectrum of quantum dots. The spectrum shift is consistent across all the array of cavities. The size of the area with the modified spectrum is ~3 µm.

The typical photoluminescence spectrum modified by the presence of a cavity is shown in Figure 7c. The position of the narrow peak on top of the broad spectrum from QD photoluminescence correspond to the resonant wavelength of the cavity. The full-width half maximum (FWHM) of the resonant peak is ~1 nm, which correspond to the Q factor of ~600. This demonstrates that the photonic mode density is largely modified within the 1d nano cavity. *This is the first experimental demonstration of rintable and 'working' nanocavities suitable for fabricating nanolasers.*

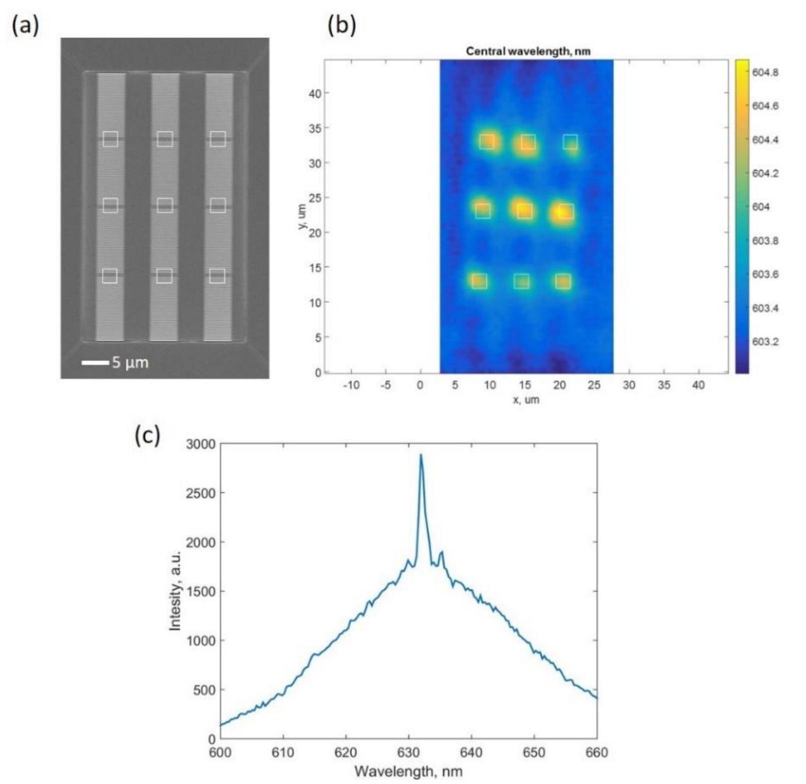

Figure 7: a) SEM image of the array of 9 cavities. b) Measured distribution of the central wavelength of the quantum dots photoluminescence spectrum. White rectangles show approximate geometrical positions of the cavities. (c) a spectrum of quantum dot photo luminescence inside a 1D cavity. The spectra exhibit a sharp peak (Q ~ 600) corresponding to the sharp resonance peak.

V. Printed Photonic Device 2: Demonstration of high refractive index light extraction layer integrated with commercial LEDs

Our materials are ceramic (TiO_2) based, a durable material suitable for applications where a harsh operating conditions are used (temperature, chemical exposure, etc). To demonstrate the versatility in the applications of our functional material, a passive photonic device was printed on top of commercial light emitting diodes (LEDs) for a better light extraction. We demonstrated improvement in the performance of a commercial LED with a thin coating of a high refractive index material. The film was mechanically texturized to increase light out-coupling efficiency of a commercial LED. A prototype device has been delivered to Air Force Scientific office Research (AFOSR).

Delivered prototype LED (450 nm) devices are:

Device 1 ('uncoated'): Commercial LED without any aBeam's polymer coating

Device 2 ('coated '): Commercial LED device with a light enhancer layer with aBeam's high refractive index polymer

aBeam's proprietary imprint polymer, '**CP2**' was used to coat the top surface of a commercial LED to improve light extraction efficiency. Measured radiometric power output of LED (Device #2, with aBeam's polymer coating) was increased by nearly **10 %** compared to Device #1 without any coating. No degradation in the materials was observed and the device maintained its optical property after extended hours of continuous illumination. ***Currently, our material is under evaluation with a major LED maker.***

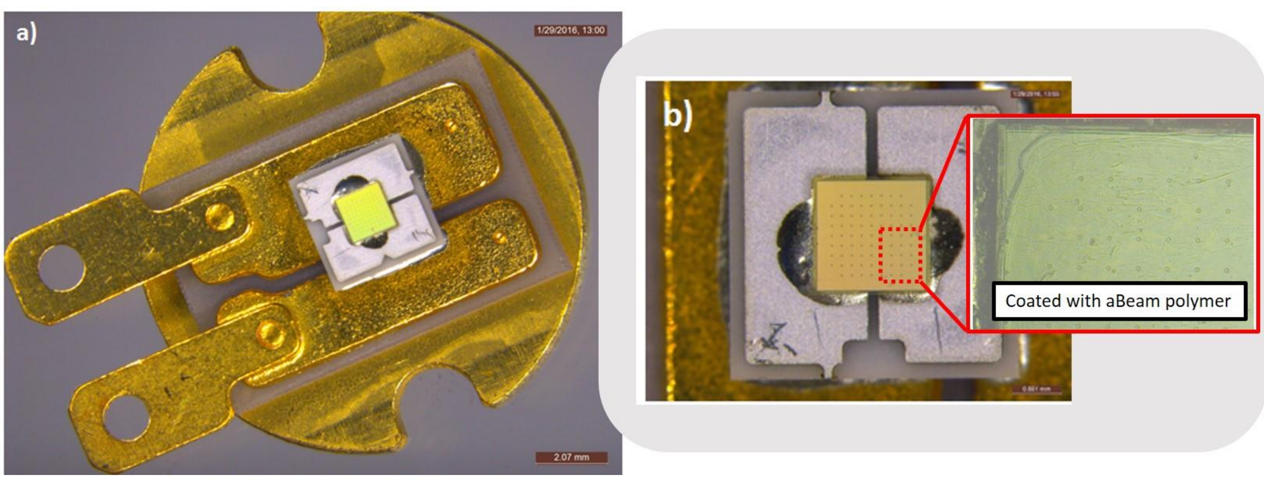

Figure 8. Optical images of LED used for experiments. a) Low magnification image of a LED device; b) a zoom in image of the LED active region, and c) LED surface coated with aBeam high refractive index polymer.

V. <u>Printed Photonic Device 3: Demonstrations of functional micro lens array fabricated imprinted with high refractive index materials</u>

We have demonstrated printed micro lens array of various dimensions into a high refractive index imprintable material developed by aBeam Technologies. The presented micro lens are printed with aBeam's '**CP4**' material with a refractive index of ***1.74*** at 590 nm. Optical characterization confirms that the printed lens are of excellent quality and produce diffraction limited focal spots. Fabrication of a micro lens array was done by UV-nanoimprint lithography as depicted in Figure 9. This process is fully scalable for printing onto a large surface area such as a screen of a mobile phone.

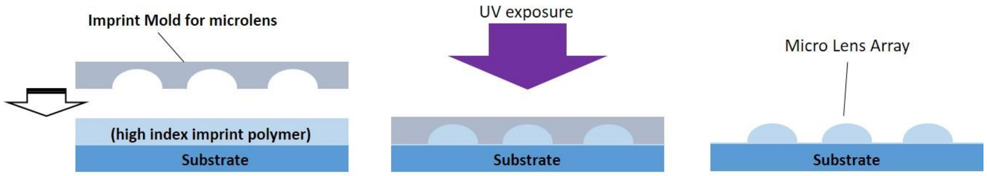

Figure 9. An illustration of the imprinting process: 1) Deposition of resist film on top of a substrate; an imprint mold is place on top. 2) UV exposure. 3) After mold removal. The pattern is transferred.

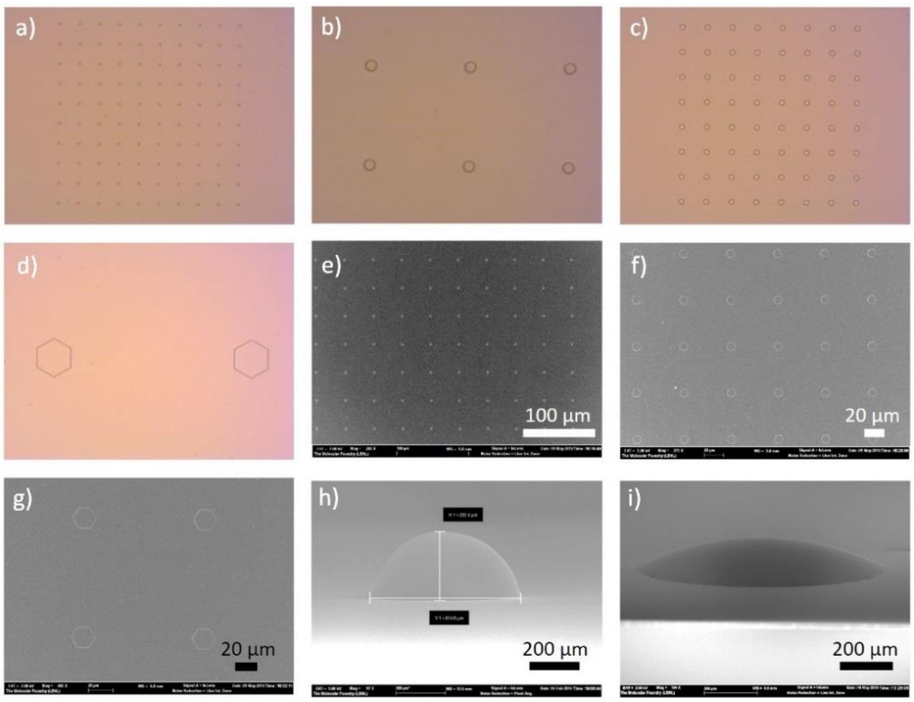

Figure 10: Images of imprinted microlens arrays with different shapes and dimensions. Optical microscope images of: (a) 3 µm diameter microlens array; (b) higher magnification image of the 3 µm diameter microlens array; (c) 8 µm microlens array; (d) 30 µm microlens array. Microlens arrays SEM top view images: (e) 3 µm diameter, (f) 8 µm diameter, (g) 30 µm diameter. SEM cross section images of large microlenses with different dimension: (h) 600 µm diameter and (i) 900 µm diameter.

Optical Characterization Imprinted Micro lens Arrays.

Optical images and scanning electron micrographs show excellent imprint quality without any visible defects.

The focusing ability of the imprinted micro lens array was characterized using a set-up shown in Figure 11a with a collimated light as an illumination source. Fig. 11b shows an array of focal spots produced by the imprinted micro lens. ***The focal spot is near diffraction limited with a full-width half-maximum (FWHM) of ~ 1 µm***, which corresponds to the predicted value (Fig. 11c &d).

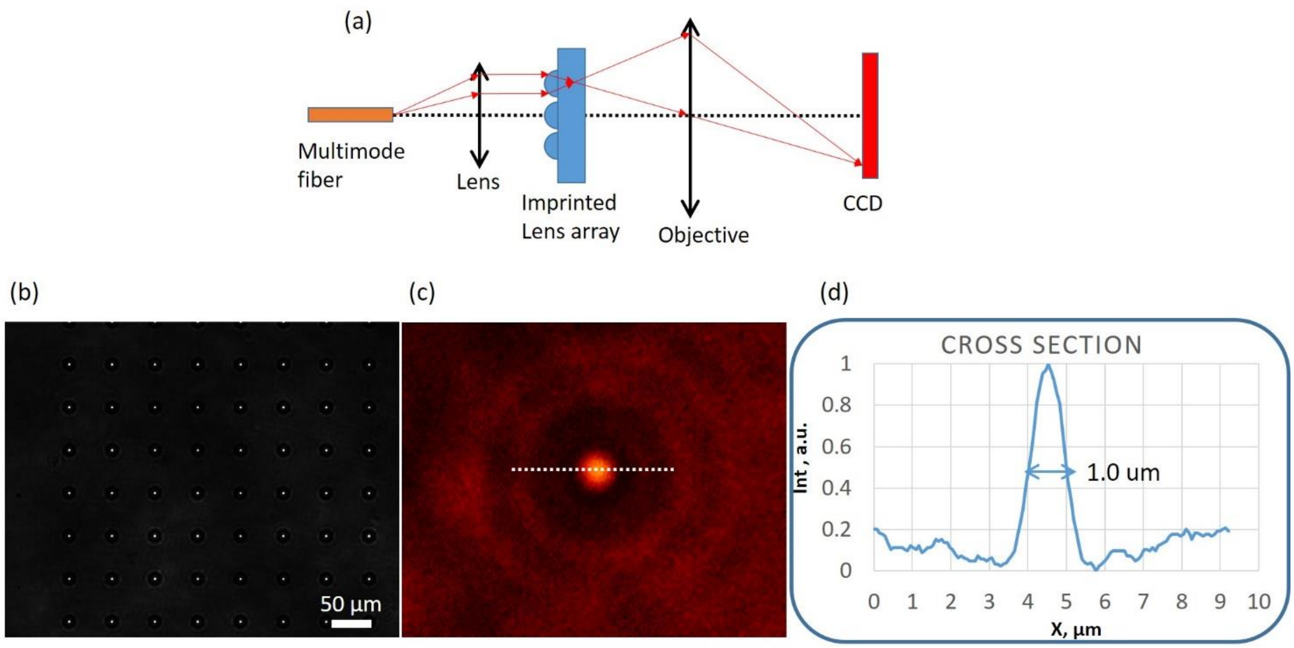

Figure 11: (a) A scheme of the optical set-up. (b) Image of focal spots produced by the imprinted microlens (3 µm diameter), (c) a zoom-in image of the focal spot produced by the printed lens, d) a cross section profile of the focal spot (white line shown in b).

Our material is for an immediate use for printing various photonic devices. Having a high refractive index is an important parameter, along with an appropriate lens design, for creating high N. A. lens. aBeam's materials have a strong advantage in this regard compared to commercially available imprint polymer in the market (n = 1.6 ~ 1.65). *Currently, two most promising high N.A. lens applications via nanoimprinting are being sought out; 1) image sensor (~3-4 µm lens diameter) and 2) light field cameras (~10-15 µm lens diameter).*

VI. Scientific and Commercial Outcome of Phase II:

Our work has been recognized by the scientific community through several publications (3 papers) as well as oral presentations at international conferences (> 10). One patent has been issued on the high refractive index material developed during the project.

- Publications resulted from the project:

1. "Printable photonic crystals with high refractive index for applications in visible light."
   Nanotechnology 2016 Mar 15;27(11):115303. Epub 2016 Feb 15.
   Giuseppe Calafiore, Quentin Fillot, Scott Dhuey, Simone Sassolini, Filippo Salvadori, Camilo Mejia, Keiko Munechika, Christophe Peroz, Stefano Cabrini, Carlos Piña-Hernandez

2. "Printable planar lightwave circuits with a high refractive index."
   Nanotechnology 2014 Aug 25; 25(32):325302. Epub 2014 Jul 25.
   Carlos Pina-Hernandez, Alexander Koshelev, Lucas Digianantonio, Scott Dhuey, Aleksandr Polyakov, Giuseppe Calafiore, Alexander Goltsov, Vladimir Yankov, Sergey Babin, Stefano Cabrini, Christophe Peroz

3. "A route for fabricating printable photonic devices with sub-10 nm resolution."
   Nanotechnology 2013 Feb 22;24(6):065301. Epub 2013 Jan 22.
   Carlos Pina-Hernandez, Valeria Lacatena, Giuseppe Calafiore, Scott Dhuey, Konstantin Kravtsov, Alexander Goltsov, Deirdre Olynick, Vladimir Yankov, Stefano Cabrini, Christophe Peroz

- Publications in preparation:

1) "Active functional nanoimprint resist with embedded Quantum Dots"
   Michaela Sainato, Carlos Pina-Hernandez, Keiko Munechika, Stefano Cabrini
   - will be submitted to Nanotechnology

2) "Printable nanolaser by nanoimprint lithography"
   Alexander Koshelev*, Carlos Pina-Hernandez*, Giuseppe Calafiore, Scott Dhuey, Christophe Peroz, Keiko Munechika, Stefano Cabrini
   - will be submitted to Nature Photonics or Nature Nanotechnology

3) "Printed flexible photonic devices by high refractive index functional resist"
   Carlos Pina-Hernandez, Alexander Koshelev, Scott Dhuey, Keiko Munechika, Stefano Cabrini
   - will be submitted to Nanotechnology

- Patent was recently issued on high refractive index material for nanoimprint lithography:

"Composition for resist patterning and method of manufacturing optical structures using imprint lithography" Patent No. : US9,298,089 B1 (Issued on March 29, 1016)

- Currently, we are on the 2^{nd} year of the contracted project for the potential licensing and production by a major chemical company.

1.

1. Report Type

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PRINTABLE INTEGRATED
PHOTONIC DEVICES

Grant/Contract Number
AFOSR assigned control number. It must begin with "FA9550" or "F49620" or "FA2386".

FA9550-14-C-0020

Principal Investigator Name
The full name of the principal investigator on the grant or contract.

Keiko Munechika

Program Manager
The AFOSR Program Manager currently assigned to the award

Gernot Pomrenke

Reporting Period Start Date

01/09/2014

Reporting Period End Date

01/08/2016

Abstract

A novel low-cost printing technology for manufacturing passive and active printable photonic components were developed based on nanoimprint lithography. Functional imprint materials were developed as well as the process to achieve state-of-the-art imprint processes with nearly zero-shrinkage in all dimensions and down to 10 nm resolution. A revolutionary type of imprint resist integrated with colloidal quantum dots (QDs) as optical gain media were developed for printing active photonic devices. Several types of printable photonic devices were successfully demonstrated including micro lens array, light extraction layer on the commercial LEDs, and revolutionary printable nanocavies for low-cost and ultra-small nanolasers.

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Archival Publications (published) during reporting period:

1. "Printable photonic crystals with high refractive index for applications in visible light."
Nanotechnology 2016 Mar 15;27(11):115303. Epub 2016 Feb 15.
Giuseppe Calafiore, Quentin Fillot, Scott Dhuey, Simone Sassolini, Filippo Salvadori, Camilo Mejia, Keiko Munechika, Christophe Peroz, Stefano Cabrini, Carlos Piña-Hernandez
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Changes in research objectives (if any):

Change in AFOSR Program Manager, if any:

Extensions granted or milestones slipped, if any:

AFOSR LRIR Number

LRIR Title

Reporting Period

Laboratory Task Manager

Program Officer

Research Objectives

Technical Summary

Funding Summary by Cost Category (by FY, $K)

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Report Document

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